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Microwave Properties of Low Density (CH)_x / *L* *X*

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MICROWAVE PROPERTIES OF LOW-DENSITY (CH)_x

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ABSTRACT

The results of an initial study of the microwave properties of low density foamlike (CH)_x are reported for intermediate dopant levels. The electrical conductivity and dielectric constant are obtained at microwave frequencies, and the results are analyzed in terms of effective medium theory. The possibility of utilizing the doped foamlike (CH)_x as an efficient microwave absorber is discussed.

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A

The synthesis of polyacetylene, $(CH)_x$, with variable density through the use of a gel as an intermediate step was recently reported.¹ The foamlike material and pressed-film samples have densities ranging from that of the as-grown film to values more than an order of magnitude smaller. The fibril morphology of $(CH)_x$ is preserved although the characteristic fibril size is larger (ca. 600-800 Å) in the lower density forms. The lower density materials simply consist of fibrils at a smaller volume filling fraction, f ; for the foamlike material with density of 0.04 gm/cm³, $f \approx 3 \times 10^{-2}$. The resulting doped and undoped $(CH)_x$ polymers can be viewed as effective media in which the electrical properties are determined by the doping level and the volume filling fraction of conducting fibrils.

In this paper we report the results of an initial study of the microwave properties of the low density foamlike material at intermediate dopant levels. The electrical conductivity (σ) and dielectric constant (ϵ) are obtained at microwave frequencies, and the results are analyzed in terms of effective medium theory. The possibility of utilizing the doped $(CH)_x$ foamlike material as an efficient microwave absorber is discussed.

Low density foamlike $(CH)_x$ was synthesized for these studies using the techniques described earlier. The density of the samples used in the microwave experiments was about 0.04 gm/cm³ as compared to 0.4 gm/cm³ for as-grown films. Since the floatation density is 1.2 gm/cm³, $f \approx 3 \times 10^{-1}$ for the as-grown films and $f \approx 3 \times 10^{-2}$ for the foamlike material.

The low density $(CH)_x$ was isomerized by heating at $180^\circ C$ for ca. 2 hours to insure that the samples were in the thermodynamically stable trans isomeric form. Prior to use, the samples were kept under a static vacuum after being pumped to 2×10^{-5} torr.

To measure the microwave conductivity and dielectric constant, waveguide voltage standing wave ratio (VSWR) and cavity perturbation techniques were utilized at 33 GHz and 10 GHz respectively. For each doping level, three samples were cut: a piece to fit into a Q-band waveguide, a sample to be mounted for four-probe dc conductivity measurements to allow in situ monitoring of the doping, and a third small reference sample. The two unmounted samples were weighed and then placed in a platinum cage attached to the back-side of the four-probe apparatus. The entire system was made of glass with platinum wiring; electrical contact to the monitor sample was made with Electrodag.

The iodine doping was carried out by passing a slow flow of dry N_2 through an iodine filled tube into the doping vessel containing the $(CH)_x$ samples and then out to traps and subsequent venting. We found that the position of samples in the doping vessel was important. If one sample was closer to the input of iodine bearing N_2 , it was doped to a higher level than samples lower down in the vessel. Further, if the flow of N_2 was too high, the outside surfaces were doped to a higher level than the interior. We thus kept the flow rate at less than 0.01 standard cubic feet per hour (scf/hr)

for $y < 0.015$ and at about 0.03 scf/hr for y up to 0.03. Under such conditions, the doping was verified to be uniform by sectioning samples after doping and checking the electrical properties. Samples for microwave measurements were weighed after doping to determine the iodine uptake and precise dopant concentration.

For VSWR measurements, the sample was placed into a section of waveguide terminated with a moveable short. The shorted section was placed on the output side of a slotted line, with the input side connected to a sweep oscillator, isolator, wavemeter, and a precision calibrated attenuator. The VSWR was determined by using the attenuator to maintain a constant voltage at the detector on the slotted line; the position of the VSWR minimum was read directly from the slotted line. Depending on conductivity and the length of the samples two possible conditions can result: either the microwave field could penetrate through the sample and be reflected from the short at the back surface, or the sample could be sufficiently strongly absorbing that reflection from the back surface was insignificant. In the first case, the single impedance method described by Roberts and Von Hippel² was used. This method requires the solution of a complex transcendental equation and thus has an infinite number of discrete solutions for σ and ϵ . One must either carry out measurements as a function of sample length or have independent information on the magnitudes to determine which branch to use. Typically, the values of

ϵ change greatly from branch to branch, whereas σ is less sensitive.

In the second case, the sample is treated as a semi-infinite slab and the single reflection problem is solved in terms of the complex dielectric function.³ Since the value obtained for ϵ is very sensitive to the precise shift in position of the wave pattern when terminated with the sample as compared with when terminated by the short, the front reference plane of the sample must be very exactly determined (an uncertainty of 0.5 mm leads to an order of magnitude error in ϵ). Thus, the roughness in the cut sample surface limits the accuracy in ϵ . Again, the value of σ is relatively insensitive to such effects and thus is determined with higher accuracy.

For the cavity perturbation measurements, a small needle shaped sample was subsequently cut from the initial waveguide sample. The method used was that described by Buravov and Shchegolev.⁴ A small ellipsoidal sample is placed in the center of a transmission cavity, and the cavity resonance frequency shift and change in half width are used to determine σ and ϵ . The sample volume and depolarization factor (η) are determined from the sample dimensions ($\eta = \frac{bc}{a^3} [\ln(\frac{4a}{b+c}) - 1]$ where a , b and c are the three orthogonal semi-axes).

In figure 1, the results of the different methods for determining the conductivity of the sample are plotted on a semi-log scale. The three independent measurements on the foamlike samples, four-probe dc, VSWR, and cavity perturbation, were in generally good agreement. Variations are

probably the result of residual variations in doping level and uncertainties in the precise sample dimensiona (particularly in the determination of η).⁵

The cavity measurements for the $y = 0.027$ sample at room temperature could not be directly evaluated due to the strong absorption and subsequent reduction in Q . We therefore cooled this sample to 100 K and estimated the room temperature result by multiplying the 100 K value by the factor $\sigma_{(RT)}/\sigma_{(100 \text{ K})}$ for iodine doped $(\text{CH})_x$ films at the same dopant concentration. In addition, we have plotted in Figure 1, the values of $\langle\sigma\rangle$ appropriate to the foamlike material using the values obtained from $(\text{CH})_x$ films (σ_f) and scaling the results according to the simple effective medium equation $\langle\sigma\rangle \approx (\frac{1}{10})\sigma_f$ where the factor $(\frac{1}{10})$ comes from the ratio of filling factors.

Fig. 2 shows the corresponding dielectric constant data. The values of ϵ were determined directly from analysis of the cavity perturbation data. The VSWR results were independent, but used the cavity perturbation results to determine the correct branch. Reliable dielectric constant data could not be obtained from the semi-infinite slab method due to front surface irregularities as described above. In addition, we have plotted in Figure 2 the values of $\langle\epsilon\rangle$ appropriate to the foamlike material using the data obtained by Mihaly et al. for $(\text{CH})_x$ films (ϵ_f) and scaling the results according to the simple effective medium equation $\langle\epsilon\rangle = 1 + \frac{1}{10}(\epsilon_f - 1)$ where again the factor $(\frac{1}{10})$ arises from the ratio of filling factors.⁶

As can be seen from Figures 1 and 2 the microwave conductivity and dielectric constant scale approximately with the density (or filling factor).

The agreement between the VSWR results and the cavity perturbation results independently implies that the low density material can be treated as an effective medium. The VSWR experiment measures the properties of the macroscopic medium that terminates the line, whereas the cavity measurement explicitly integrates over the volume of the sample and over the depolarization field induced by the presence of the dielectric. The volume used was the total volume, which is about 30 times the actual volume of $(CH)_x$ fibrils. Moreover, the depolarization factor was calculated assuming a single three-axis ellipsoid with dimensions equal to those of the piece of foamlike $(CH)_x$, implying no significant charge buildup other than at the macroscopic surface of the sample.

The skin depth (δ) at 30 GHz was calculated for the foamlike polymer as a function of dopant concentration using the full expression

$$\delta = \frac{c\sigma}{\omega\sqrt{\epsilon}} \left\{ \frac{2}{\left[1 + \left(\frac{4\pi\sigma}{\omega\epsilon} \right)^2 \right]^{\frac{1}{2}}} - 1 \right\}^{\frac{1}{2}} \quad (1)$$

and the solid lines of Figures 1 and 2 for σ and ϵ . The results are plotted in Figure 3. An experimental estimate of the skin depth was obtained using the VSWR apparatus. Starting with the sample length (l) in the infinite slab limit ($l \gg \delta$) the sample was successively sliced away until the effect of the moving short could be quantitatively detected. In this manner, for example, we determined that the skin depth for $y = 0.021$ was approximately

2 mm in satisfactory agreement with the results of Fig. 3.

In order to obtain a quantitative description of the electromagnetic properties, we model the variable density polymer as a composite effective medium, consisting of the $(CH)_x$ fibrils in a matrix. The composite medium problem was solved for isotropic spheres by Maxwell-Garnett and extended to more complex configurations by many authors.⁸ Tanner et al.⁹ considered the case of randomly oriented ellipsoids with highly anisotropic properties ($\epsilon_{||} \gg \epsilon_{\perp}$). The complex effective medium dielectric function for such a system is written

$$\langle \epsilon \rangle = \epsilon_m + \frac{\frac{1}{3}f \epsilon_m^i (\epsilon^i - \epsilon_m)}{\epsilon_m + g(1 - \frac{1}{3}f)(\epsilon^i - \epsilon_m)} \quad (2)$$

where ϵ_m is the dielectric constant of the matrix (for air $\epsilon_m = 1$), ϵ^i is the intrinsic complex dielectric constant of the particles of interest, f is the fractional volume filling factor, and g is the depolarization factor of the ellipsoidal particles. In our case, we identify the ellipsoidal particles with the $(CH)_x$ fibrils. The factor $g(1 - \frac{1}{3}f)(\epsilon^i - \epsilon_m)$ in the denominator of eq. 2 represents the reduction in internal field due to charge build-up at the boundaries. Since the fibrils are interconnected, there is no significant charge build-up except at the macroscopic boundaries of the sample. This was verified in the cavity perturbation experiments described above. Thus we take the $g \rightarrow 0$ limit and obtain

$$\begin{aligned} \text{Re}\langle\epsilon\rangle &\equiv \langle\epsilon_1\rangle = \epsilon_m + \frac{1}{3}f(\epsilon_1^i - \epsilon_m) \\ \text{Im}\langle\epsilon\rangle &\equiv \langle\epsilon_2\rangle = \frac{1}{3}f\epsilon_2^i \end{aligned} \quad (3)$$

where we have assumed ϵ_m to be real. For $\epsilon_m = 1$, eq. 3 is in agreement with the results of Figures 1 and 2.

A potentially useful application for this variable density conducting polymer is as a microwave absorbing material. Ideally such a material should have $\langle\epsilon_1\rangle$ close to unity so that reflection is minimized and determined by the conductivity of the medium. As shown above, these conditions are fulfilled for the foamlike $(\text{CH})_x$.

The general expression for the reflectance (normal incidence) of microwaves from a surface in free space is given by

$$R = \frac{(n-1)^2 + k^2}{(n+1)^2 + k^2} \quad (4)$$

- where $(n+ik)$ is the complex index of refraction and $(\langle\epsilon_1\rangle + i\langle\epsilon_2\rangle) = (n+ik)^2$. Minimizing R with n as the free parameter we obtain optimum conditions $n^2 = 1 + k^2$ so that $\epsilon_1 = n^2 - k^2 = 1$ and $\epsilon_2 = 2nk = 2k\sqrt{1+k^2}$. Under these conditions one has minimum reflectance

$$R_{\min} = \frac{(1+k^2) - (1+k^2)^{\frac{1}{2}}}{(1+k^2) + (1+k^2)^{\frac{1}{2}}} \quad (5)$$

and R_{\min} is a function of k only. The power absorption coefficient is

given by $\alpha = 2(\omega/c)k$ so that the skin depth is $\delta = (c/\omega k)$. Thus under optimum conditions ($\epsilon_1 = 1$), the reflected power and skin depth are directly related

$$\delta = \frac{\lambda_0/2\pi}{\left[\left(\frac{1+R_{\min}}{1-R_{\min}} \right)^2 - 1 \right]^{\frac{1}{2}}} \quad (6)$$

$$R_{\min} = \frac{\left[1 + \left(\frac{\lambda_0}{2\pi\delta} \right)^2 \right] - \left[1 + \left(\frac{\lambda_0}{2\pi\delta} \right)^2 \right]^{\frac{1}{2}}}{\left[1 + \left(\frac{\lambda_0}{2\pi\delta} \right)^2 \right] + \left[1 + \left(\frac{\lambda_0}{2\pi\delta} \right)^2 \right]^{\frac{1}{2}}} \quad (7)$$

where λ_0 is the free space wavelength.

For microwave absorber applications, it would be desirable to have a relatively thin coating capable of absorbing, for example, 90% of the incident microwave power. For $\lambda_0 = 1$ cm (35 GHz), $R_{\min} = 10\%$ corresponds to $\delta = 2$ mm using eq. 6 and 7.

For doped polyacetylene, we can use the values of σ and ϵ from Figures 1 and 2 to calculate δ (eq. 1) and the free-space (normal incidence) reflection coefficient R (eq. 4). The results are shown in Figs. 3 and 4. As can be seen from the figures, foamlike $(CH)_x$ doped in the range $y \sim 0.02$ is close to the optimum conditions as a microwave absorber. Moreover numerical estimates indicate that using a dielectric filler with $\epsilon_m < 2$ could lead to improved mechanical properties and good adhesion without seriously

degrading the electromagnetic properties of such a coating.

We note finally that since the quantity $(\lambda_0 / \delta) \propto \omega^{-\frac{1}{2}}$ as ω increases.

Thus, the reflectance decreases, but the absorption coefficient increases. The resulting coating would be broad band with better performance at higher frequencies.

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FIGURE CAPTIONS

Figure 1. - The microwave conductivity of foamlike $(CH)_x$ doped with iodine; $(CHI)_{y/x}$

- dc four probe
- cavity perturbation (10 GHz)
- △ VSWR (30 GHz)

The solid curve represents $\langle \sigma \rangle = \frac{1}{10} \sigma_f$ where σ_f corresponds to the values obtained from $(CH)_x$ films and the factor $(\frac{1}{10})$ comes from the ratio of filling factors (see text).

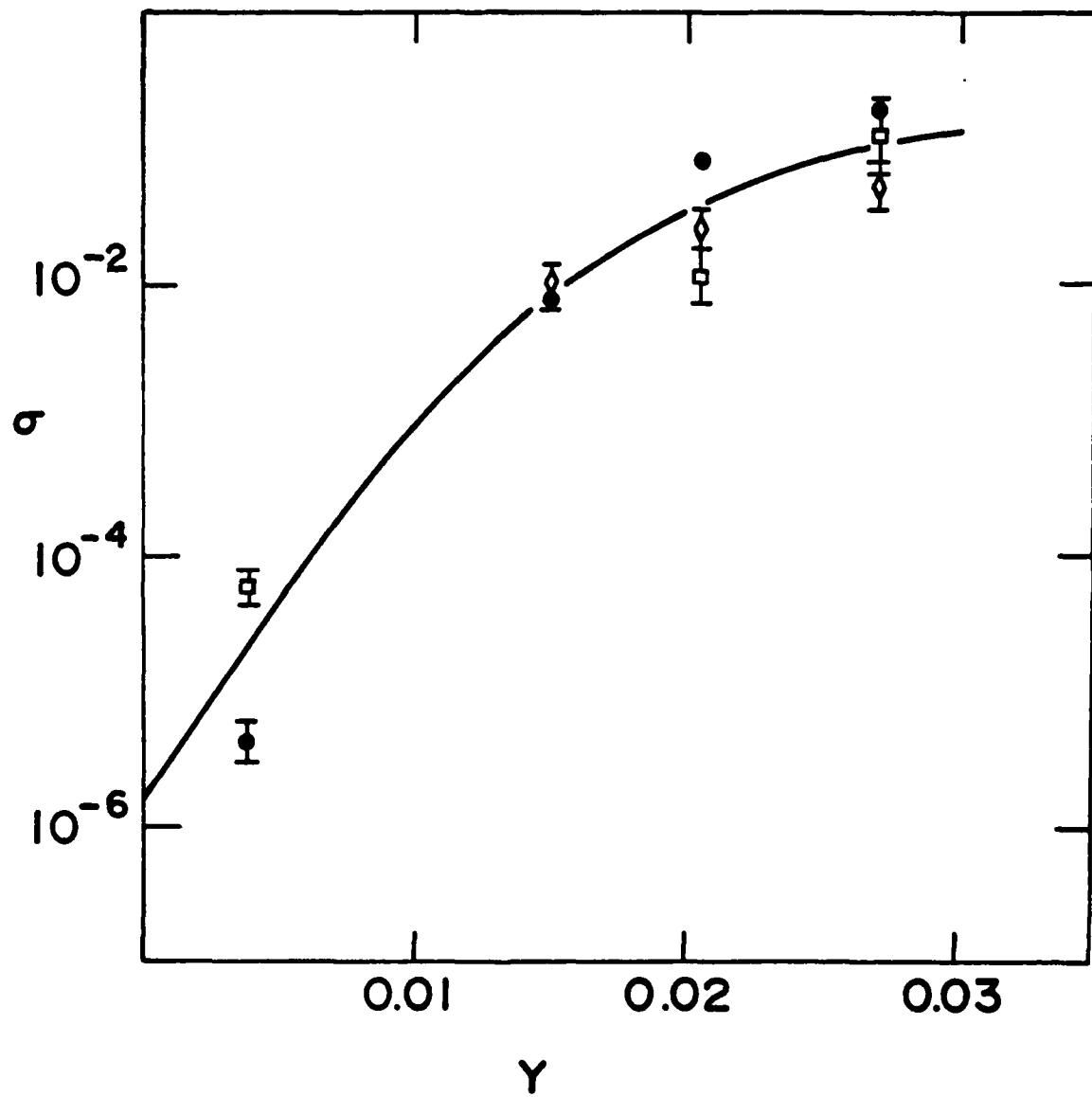
Figure 2. - The microwave dielectric constant of foamlike $(CH)_x$ doped with iodine; $(CHI)_{y/x}$

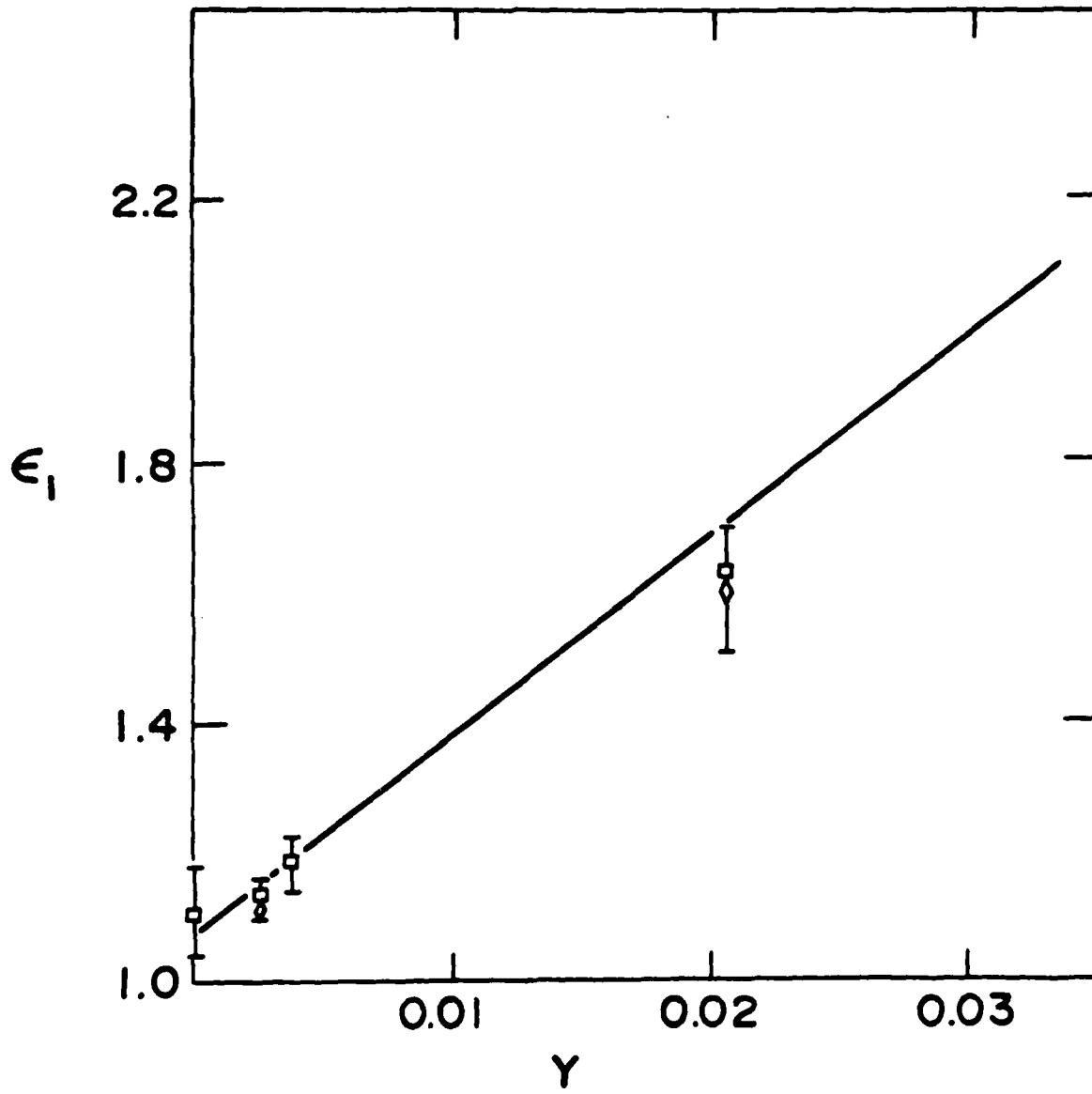
- cavity perturbation; (10 GHz)
- △ VSWR (30 GHz)

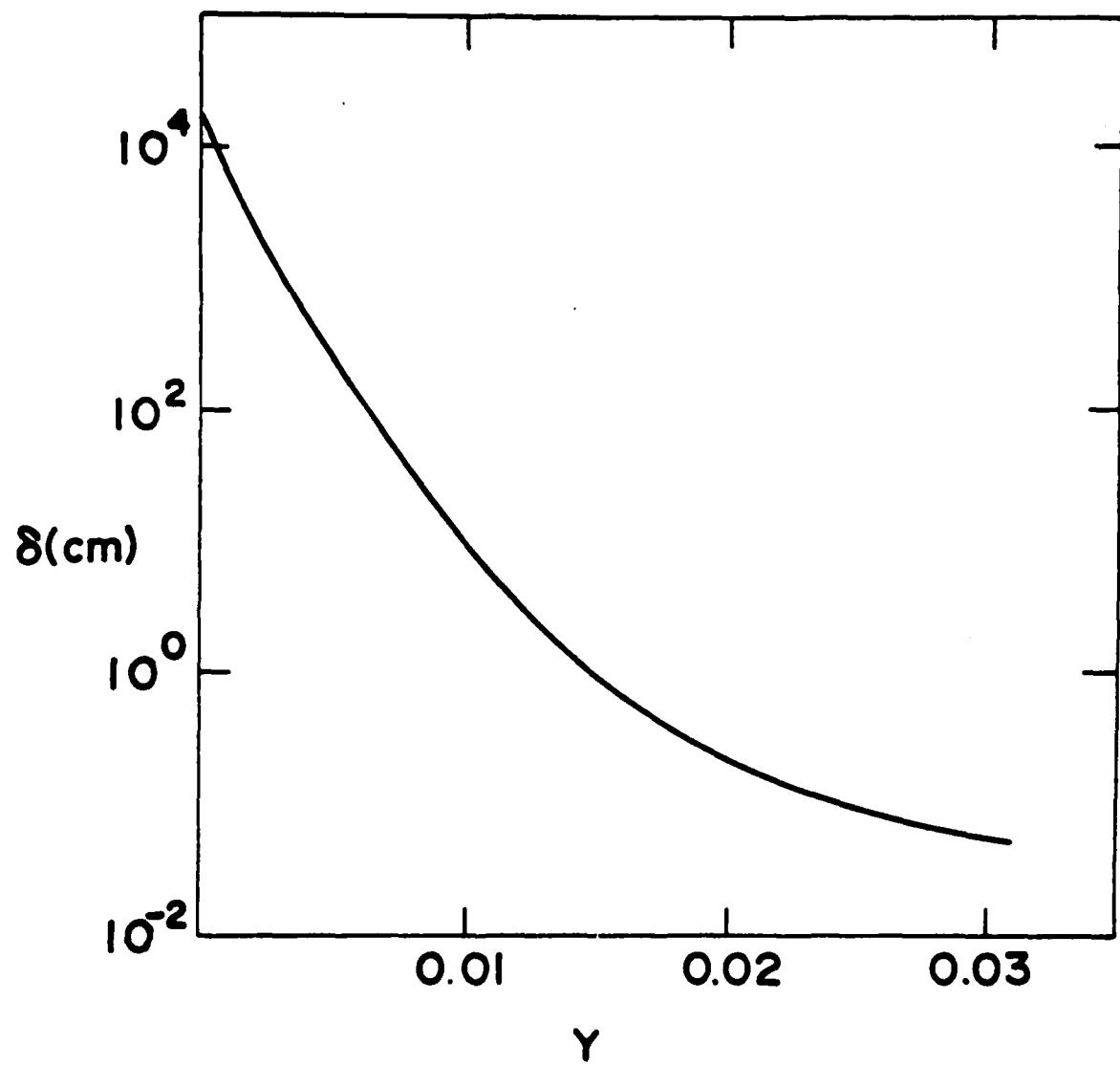
The solid line represents $\langle \epsilon \rangle = 1 + \frac{1}{10} (\epsilon_f - 1)$ where ϵ_f corresponds to the values obtained from $(CH)_x$ films and the factor $(\frac{1}{10})$ comes from the ratio of filling factors (see ref. 6).

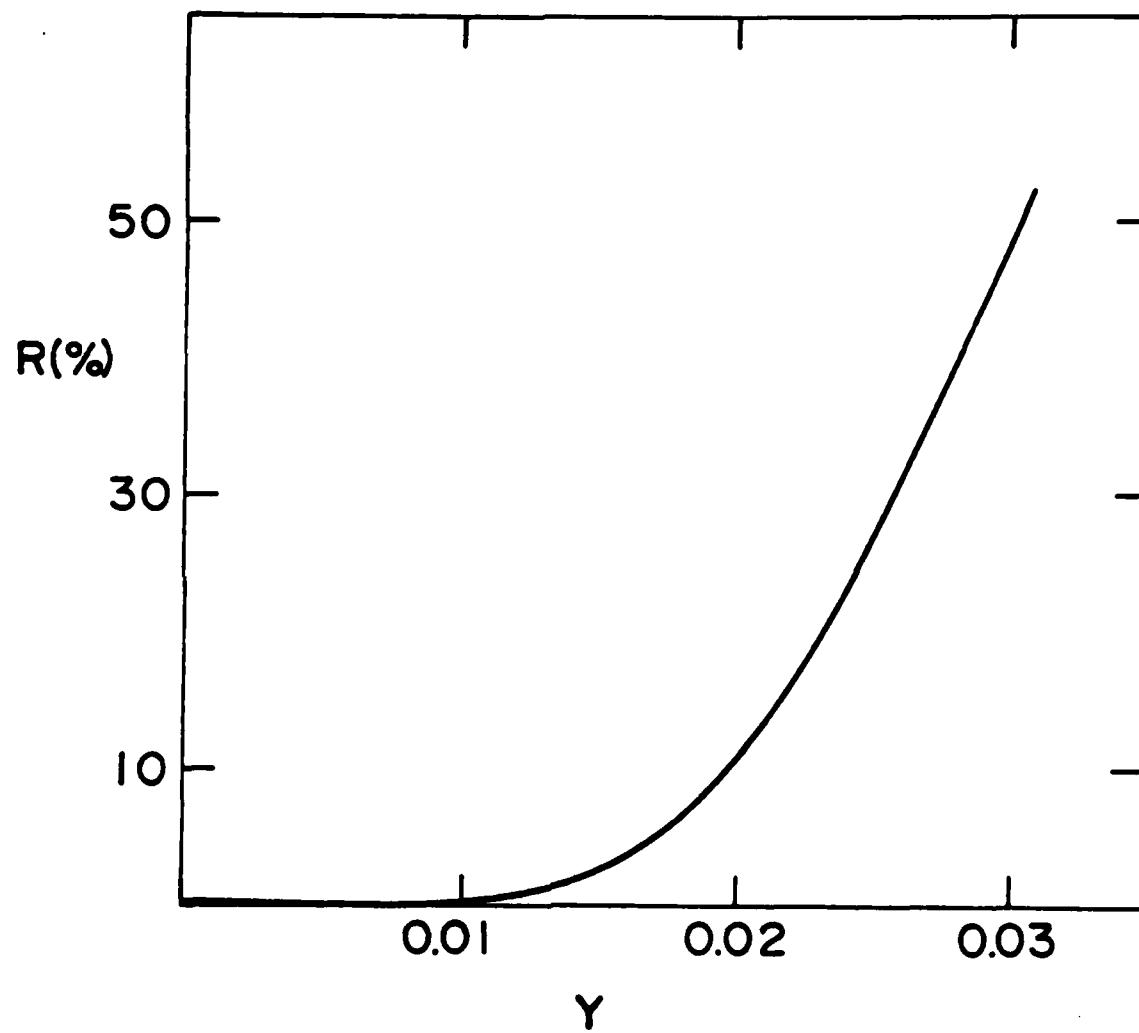
Figure 3. - Calculated skin depth δ vs. concentration y for foamlike $(CHI)_{y/x}$ using eq. 1 and the data of Figs. 1 and 2.

Figure 4. - Calculated reflection coefficient vs. concentration y for foamlike $(CHI)_{y/x}$ using eq. 4 and the data of Figs. 1 and 2.









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